

PATENTS

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant(s): Donald R. Huffman

Examiner:

Serial No.: 08/236,933

Art Unit:

Filed: May 2, 1994

Docket: 7913ZAZY

For: NEW FORM OF CARBON

Assistant Commissioner for Patents
Washington, DC 20231DECLARATION OF Harold W. Kroto UNDER 37 C.F.R. §1.132

Sir:

I, Harold W. Kroto, Ph. D., declare and say as follows:

1. I am the Royal Society Research Professor in the School of Chemistry and Molecular Sciences at the University of Sussex, Brighton, United Kingdom. I have attached for the convenience of the United States Patent Office a copy of my curriculum vitae as Exhibit 1, which describes my credentials and demonstrates my expertise in the area of fullerenes.

2. I have reviewed the above-identified application, the Preliminary Amendment therein and the following reference documents, which I understand to be cited in support of a rejection of the present application.

1. an article by K.S. Day, et al., Nature Physical Science 1973, 243, 50-51.

2. an article by Iijima, et al., in J. Phys. Chem. 1987, 91, 3466-3467. ("Iijima et al".).

3. Translation of Russian Patent No. 1,587,000.

4. U.S. Patent No. 2,957,756 to R. Bacon.

5. an article by Kappler, et al., in J. App. Phys., 1979, 50, 308-316.

3. The application teaches in clear detail to the skilled artisan the preparation of fullerenes, including C_{60} , in quantities that were never recognizably achieved before the discovery by Huffman and Kratschmer described in the application. Specifically, the application describes methods for the production of C_{60} and C_{70} in macroscopic amounts, i.e., amounts that could be seen with the naked eye (inherently at least 10^{18} molecules of product). That discovery for the first time permitted the researchers to confirm the existence and structure of these materials, including subjecting them to general testing of their detailed properties and characteristics, which had theretofore only been projected based upon educated speculation and calculation, grounded upon circumstantial evidence of their existence.

4. I am intimately familiar with the literature concerning and was personally involved in the search for C_{60} and the greater fullerene family: for convenience, one may refer particularly to our review of the literature through 1990 described in an article entitled " C_{60} Buckminsterfullerene, in Chem. Rev. 1991, 1231-1235 attached hereto as Exhibit 2 and for my personal involvement in the research effort in my article entitled " C_{60} : Buckminsterfullerene, the Celestial Sphere that Fell to Earth" in Angewandte Chemie I.E.E. 1992, 31, 111-129, attached as Exhibit 3.

5. I believe it is fair to say that I am among the recognized experts in the subject of fullerenes, and that I was quite cognizant of the state of the art in 1990, and of the early attempted preparation and identification of fullerenes, especially C_{60} and C_{70} .

6. I am familiar with the work of Huffman and Kratschmer on this subject having closely followed their research as described in the Angewandte Chemie article referred to above and attached as Exhibit 3.

7. I am also familiar with the methods described in the above-identified patent application of Huffman and Kratschmer and utilize their principles regularly in producing quantities of C_{60} for research purposes in our laboratories,

8. In my professional opinion the methods for producing the fullerenes, including C_{60} , are described in the application in such manner as to enable one skilled in the art to make and use the same.

9. In my professional opinion, the reference documents listed in paragraph 2 hereinabove, taken individually or collectively, do not teach nor do they claim to teach methods for the production of fullerenes, including C_{60} ; nor is there provided evidence of the production of any such product. Specifically, it cannot be stated that there is any reliable scientific evidence of the formation of C_{60} or C_{70} in any of the references, and no assertion is made that quantities of C_{60} or C_{70} were made. In fact, any such assertion would be entirely speculative and unsupported; to my knowledge, no researcher had proven possession of C_{60} or C_{70} prior to Huffman and Kratschmer.

While Iijima et al alleges that they saw a molecule of C_{60} in the middle of a carbon particle this conclusion is similarly entirely speculative and unsupported by the evidence. Furthermore, Iijima et al did not report in that article a methodology capable of producing and isolating fullerenes in

10. The realization by Huffman and Kratschmer of macroscopic quantities of fullerene and the isolation and characterization of C_{60} and C_{70} by the methods described in the above-identified application is recognized by the knowledgeable scientific community as a long awaited and much needed breakthrough; it was surprising that relatively high yields of fullerene such as C_{60} could be achieved by these methods, as it was expected that no more than $< 1/10000$ parts of target molecules would exist in the soot product and that it would require very sophisticated equipment to isolate quantities of material required to establish and confirm the existence of the products. The difficulties that existed in the quest for C_{60} are well elaborated in the article entitled "Fullerenes" by Robert F. Curl and Richard E. Smalley, printed in Scientific American, Oct. 1991, pp. 54-62 attached hereto as Exhibit 4.

11. Although the discovery described in the Huffman and Kratschmer application may seem simplistic to the uninformed, especially in hindsight, their discovery was quite remarkable. This is readily appreciated if one considers the historical perspective. Ever since the detection of C_{60} by the collaborative efforts of the Smalley and Kroto groups in 1985, as described in the article in Nature, 1985, 318, 162-163, attached hereto as Exhibit 5, experts, such as Drs. Smalley and myself, both together and separately worked to prepare fullerenes on a larger scale. For five long years, many attempts were tried, but each were unsuccessful. Finally, to my knowledge, one group, Huffman and Kratschmer, were the first to find a methodology capable of producing and isolating fullerenes, such as C_{60} , in macroscopic amounts. This methodology is described in their application and satisfied a long felt need in this area.

12. Furthermore, one should not underestimate the significance of their discovery. For the first time, scientists were able to produce and work with samples of fullerenes. They were able to confirm the theoretical predictions about fullerenes and continue to explore new properties of same. Their discovery spawned enormous scientific interest. As a consequence, innumerable investigations and studies relating to fullerenes were conducted, generating more than four thousand publications on the subject. In short, I cannot emphasize enough that their discovery revolutionized the area of fullerenes.

13. I have been requested as well to examine the claims presented by applicants Huffman and Kratschmer. I am not qualified in the law as to the interpretation of claims; but as a scientist knowledgeable in this art, I find the qualifying terms to be aptly descriptive of the methods described and the products produced in the above-identified application, consistently with scientific usage at the time the application was filed.

14. I further assert that the term "macroscopic" aptly and correctly characterizes the breakthrough made by Huffman and Kratschmer in permitting isolation and characterization of the fullerenes C_{60} and C_{70} , in that the term expressly denotes that which can be seen (and therefore tested); that usage is consistently employed in my papers and reviews on the subject entirely independently of Huffman and Kratschmer.

15. In my professional judgement, the above-identified application adequately teaches to the skilled artisan how to make macroscopic amounts of the fullerenes including C_{60} and C_{70} ; furthermore, there is ample evidence in the application that

Huffman and Kratschmer had in their possession macroscopic amounts of these products.

16. I have been among those who sought an appropriate name for this family of often co-produced structurally related material and based upon structure and the historical connection with the geodesic dome structures of Buckminster Fuller, I introduced the name of fullerenes for these molecules in 1987 which was later accepted by the scientific community fullerene by about 1990, and this has become the accepted formal name for these materials, e.g., [60] fullerene and [70] fullerene. I refer in particular to the definition I prepared for McGraw-Hill appearing in McGraw Hill Concise Encyclopedia of Science & Technology, 3rd ed. p. 819 (1994).

17. In summary, I am pleased to lend support to the applications of Huffman and Kratschmer for patent protection; as a researcher in the quest for C_{60} I can keenly appreciate the significance of the defining events reflected in the present application; I can, from my own experience, state with confidence that despite our circumstantial evidence of the existence of these molecules, the inevitable speculation and calculations of properties, and our own convictions, given our knowledge at the time, it was by no means predictable nor obvious to one skilled in the art that fullerenes, such as C_{60} or C_{70} , would be recovered in macroscopic quantities by the methods described by Huffman and Kratschmer in the above-identified application, nor to the best of my knowledge, had such results been claimed.

18. I further declare that all statements made herein of my own knowledge are true and that all statements made on information

statements and the like so make are punishable by fine or imprisonment or both under section 1001, Title 18 of United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Dated: 9 June 1995

Harold W. Kroto, Ph. D.